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IS 10085 (2003): Chemical Analysis of Zircon Flour or Sand  
[MTD 13: Ores and Raw Materials]



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भारतीय मानक  
जीरकन आटा अथवा रेत का रासायनिक विश्लेषण  
( पहला पुनरीक्षण )

*Indian Standard*

CHEMICAL ANALYSIS OF ZIRCON FLOUR OR SAND  
( *First Revision* )

ICS 73.060.99

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industrial Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1982. It covered the methods for determination of loss on ignition and of the oxides of zirconium, silicon, iron and titanium in zircon flour or sand. Further, these methods could be used for similar materials having zirconia content of not more than 80 percent.

In this revision, keeping in view the gaps generated during these years, the following changes have been made:

- a) The thiocyanate (photometric) method for determination of iron oxide has been replaced by orthophenanthroline method,
- b) The mandelic acid method for determination of zirconium oxide has been specified as referee method and an alternate 'EDTA' method has been updated,
- c) Method for determination of alumina has been incorporated,
- d) Determination of silica by gravimetric method has been updated on the basis of experiences gained, and
- e) The photometric method for determination of titania has also been updated based on the experiences gained.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off values should be the same as that of the specified value in this standard.

# Indian Standard

## CHEMICAL ANALYSIS OF ZIRCON FLOUR OR SAND (First Revision)

### 1 SCOPE

This standard describes the methods for determination of loss on ignition and of the oxides of silicon, iron, titanium zirconium and aluminium in zircon flour or sand.

### 2 REFERENCES

The following standards are necessary adjuncts to this standard.

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
265 : 1993	Hydrochloric acid ( <i>fourth revision</i> )
266 : 1993	Sulphuric acid ( <i>third revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
1811 : 1984	Method of sampling foundry sand ( <i>first revision</i> )

### 3 SAMPLING

The sample shall be drawn and prepared as per the method given in IS 1811.

### 4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

### 5 DETERMINATION OF LOSS ON IGNITION

#### 5.1 Outline of the Method

The sample is heated in a muffle furnace at 950°C and the percent loss in mass is calculated.

#### 5.2 Procedure

Transfer 1 g of the test sample to a weighed platinum crucible and heat in a muffle furnace or in the flame of burner at a temperature between 950° C and 1 000°C for half-an-hour. Cool in a desiccator and weigh. Repeat the procedure till constant mass is obtained on two successive heatings. Calculate the loss of mass of the sample.

#### 5.3 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{A \times 100}{B}$$

where

$A$  = loss of mass, in g, on ignition, and

$B$  = mass, in g, of the sample taken.

### 6 DETERMINATION OF SILICA BY THE GRAVIMETRIC METHOD

#### 6.1 Outline of the Method

The sample is fused with borax-sodium carbonate mixture and silica is determined by gravimetric method after dehydration of the decomposed sample in sulphuric acid. Boric acid is removed as methyl borate by heating with methyl alcohol and residual silica is determined photometrically.

#### 6.2 Reagents

**6.2.1 Anhydrous Sodium Carbonate and Borax, 1:1 mixture.**

**6.2.2 Dilute Sulphuric Acid, 1:1 and 1:10 (v/v).**

**6.2.3 Methyl Alcohol, 99 percent minimum.**

**6.2.4 Hydrofluoric Acid, 40 percent (v/v).**

**6.2.5 Standard Silica Solution (0.10 mg/ml)** — Fuse 0.100 g of pure silica with 2 g of sodium carbonate in a platinum crucible. Cool the melt and dissolve in 300 ml of water, containing 3-4 pellets of sodium hydroxide, in a polythene beaker. Dilute to 1 000 ml in a volumetric flask. Store in a polythene bottle.

**6.2.5.1 Standard silica solution (0.01 mg/ml)** — Take 10 ml of standard silica solution (*see* 6.2.5) in 100 ml volumetric flask. Dilute to the mark and mix well.

**6.2.6 Potassium Bisulphate, solid.**

**6.2.7 Potassium Chlorate Solution, 7 percent (m/v).**

**6.2.8 Boric Acid, 5 percent (m/v).**

**6.2.9 Potassium Permanganate, 10 percent (m/v).**

**6.2.10 Molybdic Acid** — Dissolve 25 g of ammonium molybdate in water and acidify the solution with 20 ml of sulphuric acid ( $\rho = 1.84$ ), cool and make up the volume of the solution to 250 ml and store in a polythene bottle.

**6.2.11 Tartaric Acid, 20 percent (m/v).**

**6.2.12 Reducing Mixture** — Dissolve 10.8 g of sodium sulphite in about 60 ml of hot water. Add 2 pellets of

sodium hydroxide and 0.2 g of 1 amino-2 naphthol-4 sulphonic acid, cool and make up to 100 ml.

NOTE — Prepare this solution fresh every time.

### 6.3 Procedure

#### 6.3.1 Decomposition of the Sample

Weigh accurately 0.5 g of finely ground and dried sample (105-110°C) in a platinum crucible. Add 6 g of borax-sodium carbonate mixture (1:1) and mix thoroughly. Cover the mass with the layer of 1 g more of the flux. Fuse the contents of the crucible with lid by placing it on a gas burner. Gradually increase the flame to its maximum to get a clear melt. Keep it for 10 to 15 min with occasional swirling of the crucible with a pair of platinum tipped tongs. Remove the crucible and cool.

6.3.2 Transfer the fused mass (*see* 6.3.1) from the platinum crucible to the beaker using water and 25 ml of dilute sulphuric acid (1:1). Warm the solution until disintegration of melt is complete and crush any lumps remaining in the solution with glass rod.

6.3.3 Cool the solution at room temperature, then add 25 ml of methanol and evaporate gently on a steam bath with occasional stirring with a glass rod until most of the methanol has been removed. Again allow the solution to cool slightly and add 25 ml of methanol. Evaporate on a steam bath, breaking up from time to time the crust of salts that form and hinder evaporation. When the evaporation is almost complete, heat the contents on a hot plate or sand bath to copious fuming with occasional stirring with a glass rod. Cool and add about 50 ml water and digest for 5 min on the hot plate. Add 1/4 ashless tablets on a little ashless pulp and stir thoroughly the solution.

6.3.4 Filter through medium textured filter paper and wash 5 to 6 times with warm dilute sulphuric acid (1:10) and thereafter several times with warm water until free from soluble salts. Preserve the filtrate. Transfer the precipitate with the filter paper to a platinum crucible and heat gently. When the precipitate with the filter paper becomes dry, increase the temperature and ignite slowly till the black carbon of the filter paper is white. Then ignite the residue at 1 000°C for 30 min. Cool in a desiccator and weigh. Add a few drops of water to moisten the oxide and 5 to 6 drops of dilute sulphuric acid (1:1). Then add 10 to 15 ml of hydrofluoric acid and heat on a hot plate provided with a thin asbestos sheet. Continue heating till the fuming ceases, then ignite the residue at 1 000°C for 30 min. Cool and weigh as before. The difference in the two masses represents the mass of pure silica.

6.3.5 Fuse the contents in the platinum crucible with potassium bisulphate and take with water and dilute sulphuric acid (1:1). Add to the filtrate preserved under 6.3.4 and dilute it to 250 ml (solution S).

#### 6.3.6 Recovery of Silica

6.3.6.1 Transfer 10 ml aliquot of the solution (*see* 6.3.5) to a 250 ml PTFE beaker. Add 1 ml of hydrofluoric acid and place the sample in the water bath maintaining temperature between 60 to 70°C. After 30 min, take out the beaker and rinse the lid into the beaker with distilled water. Add 0.5 ml of potassium chlorate solution (7 percent).

6.3.6.2 Add 40 ml of boric acid (5 percent) followed by a few drops of potassium permanganate solution till the solution retains a permanent pink tinge. Add 4 ml of molybdic acid and allow the solution to stand for 15 to 20 min. Now adjust the pH to  $1.25 \pm 0.05$  using dilute ammonium hydroxide and dilute hydrochloric acid. Wait for 10 min. Cool the solution to  $21 \pm 2^\circ\text{C}$ . Add 4 ml of reducing mixture. Swirl the solution after each addition and allow it to stand for 30 min. Make up the volume to 100 ml.

6.3.6.3 Transfer a suitable portion of the coloured solution to an absorption cell and take the photometric reading at 815 nm against a reference test blank solution prepared in the same way but without the sample.

#### 6.3.6.4 Test blank solution

Prepare the solution by taking all reagents and solution except the sample in accordance with the general procedure for the determination of residual silica.

#### 6.3.6.5 Calibration curve

Transfer 0, 1, 2, 3, 4 and 5 ml of the standard silica solution (*see* 6.2.5.1) to 250 ml PTFE beaker. Dilute each to 10 ml with water. Add 0.5 ml dilute sulphuric acid (1:1) and carry out the entire stages of procedure as described in 6.3.6.1 (except the addition of 10 ml of solution S), 6.3.6.2 and 6.3.6.3 using the same quantity of reagents and record the photometric readings of all the solutions against the blank (10 ml).

6.3.6.6 Convert the photometric readings of the test solution to milligrams of silica by means of the calibration curve and calculate the silica content in grams present in 250 ml of solution S (*see* 6.3.5).

### 6.4 Calculation

$$\text{Silica, percent by mass} = \frac{(A - B + C) \times 100}{D}$$

where

A = mass, in g, of crucible with lid and contents before hydrofluorization;

B = mass, in g, of crucible with lid and residue after hydrofluorization;

C = mass, in g, of silica present in 250 ml of solution S; and

D = mass, in g, of the sample taken.

## 7 DETERMINATION OF IRON OXIDE BY THE 1,10 ORTHOPHENANTHROLENE (PHOTOMETRIC) METHOD

### 7.1 Outline of the Method

Ferrous ions in the pH range of 3 to 6 forms a light orange coloured soluble complex with 1, 10 orthophenanthroline. The complex absorbs at 510 nm and is used for determination of iron spectrophotometrically.

### 7.2 Reagents

**7.2.1 Sulphuric Acid**, rd = 1.84 (conforming to IS 266).

**7.2.2 Tartaric Acid**, 50 percent (m/v).

**7.2.3 1,10 Orthophenanthroline Solution**, 0.25 percent (m/v) in hot water.

**7.2.4 Hydroxylamine Hydrochloride**, 10 percent (m/v).

**7.2.5 Stock Iron Solution** (1 ml = 1 mg  $\text{Fe}_2\text{O}_3$ ) — Weigh accurately 0.699 4 g of iron turnings (purity 99.9 percent) in a beaker and dissolve it in a mixture of hydrochloric acid and nitric acid. After complete dissolution, add 10 ml of sulphuric acid and fume to dense white fumes. Cool, add water and warm the beaker gently to dissolve the salt. Cool and transfer to 1 000 ml volumetric flask. Dilute to mark. One millilitre of this solution contains 1 mg of iron oxide ( $\text{Fe}_2\text{O}_3$ ).

**7.2.5.1 Standard iron solution** (1 ml = 0.01 mg  $\text{Fe}_2\text{O}_3$ ) — Dilute 10 ml of the standard stock solution (see 7.2.5) to 1 000 ml water.

**7.2.6 Dilute Ammonium Hydroxide**, 1:1 (v/v).

### 7.3 Procedure

**7.3.1** Transfer 10 ml of aliquot of the solution preserved as described in 6.3.5 (solution S) to a 100 ml beaker. Add 2 ml of tartaric acid followed by 2 ml of hydroxylamine hydrochloride. Adjust the pH to 4.5 using dilute ammonium hydroxide. Add 2 ml of 1.10 orthophenanthroline and make up to 50 ml. After 10 min, measure the absorbance of the solution at 510 nm using blank run through the same procedure.

### 7.3.2 Calibration Curve

**7.3.2.1** Transfer 0, 2, 4, 6, 8 and 10 ml of the standard iron solution (see 7.2.5.1) in 100 ml beakers. Using same quantity of reagents, carry out the entire stages of procedure as described under 7.3.1 and record the photometric readings of all the standard solutions against the blank.

**7.3.2.2** Convert the photometric readings of the sample to milligrams of iron by means of a calibration curve.

### 7.4 Calculation

$$\text{Ferric oxide, percent by mass} = \frac{A \times 250 \times 100}{B \times 10 \times 1\,000}$$

where

$A$  = mass, in mg, of iron oxide found in the aliquot; and

$B$  = mass, in g, of the sample taken.

## 8 DETERMINATION OF TITANIA BY THE PHOTOMETRIC METHOD

### 8.1 Outline of the Method

The yellow coloured complex produced in acid solution of the sample by the addition of hydrogen peroxide is determined photometrically at 420 nm.

### 8.2 Reagents

**8.2.1 Hydrogen Peroxide**, 20 percent (v/v).

**8.2.2 Dilute Sulphuric Acid**, 1:10 (v/v).

**8.2.3 Standard Titania Solution** (1 ml = 0.25 mg  $\text{TiO}_2$ ) — Weigh accurately 0.25 g of pure titanium dioxide and fuse with 6 g of potassium pyrosulphate in a platinum crucible. Dissolve the melt in hot dilute sulphuric acid (1:10). Dilute the solution to 1 000 ml in a volumetric flask using dilute sulphuric acid 1:10.

### 8.3 Procedure

**8.3.1** Transfer 25 ml of aliquot of the solution (S) preserved under 6.3.5 to 50 ml volumetric flask add 5 ml of hydrogen peroxide, mark up to volume.

**8.3.2** Transfer the suitable portion of the solution (see 8.3.1) in a 1 cm quartz cell and record photometric reading at 420 nm against a sample blank.

#### NOTES

- 1 Against blank prepared by using 25 ml of solution (S) diluted to 50 ml.
- 2 Hydrogen peroxide bubbles when shaken. All the care should be taken with measurement that no bubble is present in the absorption cell.

### 8.3.3 Calibration Curve

Transfer 0, 1, 2, 4 and 6 ml of the standard titania solution (see 8.2.2) to 50 ml volumetric flasks, using the same quantity of reagents, carry out the entire stages of procedure as described in 8.3.1 and 8.3.2 and record the photometric readings of all the standard solutions against the standard solution process blank.

Convert the photometric reading of the sample to milligrams of titania by means of calibration curve.

### 8.4 Calculation

$$\text{Titania, percent by mass} = \frac{A \times 250 \times 100}{B \times 25 \times 1\,000}$$



where

$A$  = mass, in mg, of titania determined in aliquot solution from the calibration curve; and

$B$  = mass, in g, of sample taken.

## 9 DETERMINATION OF ZIRCONIA BY MANDELIC ACID METHOD

### 9.1 Outline of the Method

Zirconia is precipitated with mandelic acid in hydrochloric acid medium and the precipitate is ignited to get zirconia. For this, sample is fused with borax-sodium carbonate mixture and the melt is dissolved in dilute sulphuric acid. Fumed with sulphuric acid to separate silica. The clear filtrate is precipitated with mandelic acid, under conditions described.

### 9.2 Reagents

#### 9.2.1 Ammonium Hydroxide Solution

#### 9.2.2 Dilute Hydrochloric Acid, 1:1 (v/v).

#### 9.2.3 Mandelic Acid, 16 percent (m/v)—Dissolve 16 g of mandelic acid in warm water and dilute to 100 ml.

#### 9.2.4 Mandelic Acid Wash Solution (2 percent)—Dissolve 10 g of mandelic acid in warm water. Add 25 ml of hydrochloric acid and dilute to 500 ml.

### 9.3 Procedure

Transfer 100 ml aliquot of the solution preserved under 6.3.5 (solution S) to a 250 ml glass beaker. Boil the solution. Add ammonium hydroxide solution in slight excess to precipitate hydroxides. Filter the solution through medium textured filter paper. Reject the filtrate. Transfer the precipitate to the same beaker using 10 ml of distilled water. Add 40 ml of hydrochloric acid (1:1). Boil the solution. Precipitate zirconia using 50 ml of hot mandelic acid solution (16 percent). Cover the beaker and settle the precipitate on a boiling water bath for 30 min. Filter the solution through the medium textured filter paper. Wash the precipitate with hot wash solution (see 9.2.4). Transfer the precipitate along with filter paper into a well cleaned, dried, weighed platinum crucible. Char over a small flame and ignite on a muffle furnace, starting from low temperature to 900°C for 30 min or more. Cool in a desiccator and weigh. Repeat the ignition till constant mass is obtained on two successive heatings. Deduct the empty platinum crucible mass to get the mass of zirconia present in the aliquot.

### 9.4 Calculation

$$\text{Zirconia (ZrO}_2\text{), percent by mass} = \frac{m \times 100}{M} \times \frac{250}{100}$$

where

$m$  = mass, in g, of the  $\text{ZrO}_2$  obtained; and

$M$  = mass, in g, of the sample taken.

## 10 DETERMINATION OF ZIRCONIA BY EDTA METHOD INVOLVING BACK-TITRATION WITH BISMUTH NITRATE (ALTERNATE METHOD)

### 10.1 Outline of the Method

From an aliquot of the solution obtained by fusion of the sample with borax-sodium carbonate mixture and dissolution in nitric acid, zirconia is determined after adding excess EDTA and back titrating the excess with bismuth nitrate using thiourea indicator.

### 10.2 Reagents

#### 10.2.1 Anhydrous Sodium Carbonate and Borax Mixture, 1:1 (m/m).

#### 10.2.2 Nitric Acid, rd = 1.42 (conforming to IS 264).

#### 10.2.3 Dilute Nitric Acid, 1:1 and 1:3 (v/v).

#### 10.2.4 Hydrochloric Acid, rd = 1.16 (conforming to IS 265).

#### 10.2.5 Xylenol Orange Indicator, 0.1 percent (m/v) in water.

#### 10.2.6 Bismuth Nitrate—Dissolve 12.127 g of bismuth nitrate $[\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}]$ in dilute nitric acid. Warm to complete dissolution. Add 10 ml of excess nitric acid. Cool and make upto 1 000 ml. Standardize against EDTA solution using thiourea as indicator.

#### 10.2.7 Buffer Solution (pH 5.3)—Dissolve 10.8 g of sodium acetate with 1 ml of acetic acid in 150 ml of water and dilute to 500 ml.

#### 10.2.8 Standard Zinc Chloride Solution (0.025 M)—Dissolve by heating 0.817 g of metallic zinc in the form of pellets or globules or foils in about 200 ml of water containing 10 ml of hydrochloric acid. Cool and dilute the solution to 500 ml in a calibrated flask.

#### 10.2.9 Standard EDTA Solution (0.025 M)—Dissolve 9.30 g of disodium salt of EDTA in water and dilute to 1 litre in a calibrated flask. Standardize this solution against standard zinc chloride solution (see 10.2.8) as by the procedure given in 10.2.9.1.

#### 10.2.9.1 Pipette 25 ml of the zinc chloride solution in a 250 ml conical flask. Neutralize with ammonium hydroxide solution (1:3) in presence of one or two drops of methyl orange. Add 10 ml of buffer solution. Titrate the solution with EDTA in presence of xylenol orange.

#### 10.2.10 Tartaric Acid Solution—50 percent aqueous solution (m/v).

#### 10.2.11 Thiourea—solid.

### 10.3 Preparation of Stock Solution

Weigh accurately 0.5 g of the finely ground and dried

sample (105°C-110°C) in a platinum crucible. Add 6 g of borax-sodium carbonate mixture (1:1) and mix thoroughly. Cover the mass with a layer of 1 g more of the flux. Fuse the content of the crucible with lid by placing it on a gas burner. Gradually increase the flame to maximum to get a clear melt. Keep it for 5 min with occasional swirling of the crucible with a pair of platinum tipped tongs. Remove the crucible. Cool and place it in a 250 ml beaker containing 75 ml of dilute nitric acid (1:3). Add 25 ml of water, cover the beaker with a watch glass and place it on a hot plate. When the melt has completely dissolved, remove the crucible and the lid and wash them with water. Add the washings to the solution and boil for a few minutes. If the solution is not clear, filter through medium textured filter paper. Char and ignite the residue. Fuse it with 1 g of borax, sodium carbonate mixture, dissolve the cooled melt in a few ml of dilute nitric acid (1:3) and add the solution to the filtrate. Transfer the clear solution to 250 ml volumetric flask and dilute with water to the mark. This represents the stock solution for determination of zirconia.

#### 10.4 Procedure

Pipette out 25 ml aliquot (*see* 10.3) into a 500 ml conical flask. Add about 100 ml of distilled water. Add exactly 20 ml of standard EDTA solution. Add 2 ml of 50 percent tartaric acid and adjust the pH to 2.0 with dilute ammonium hydroxide or dilute nitric acid. Add 3 g of solid thiourea, and titrate with standard bismuth nitrate (*see* 10.2.6) to the first formation of the yellow colour. If the bismuth nitrate solution contains much free acid, it may be necessary to readjust the pH to 2.0 slightly before the end point is reached. Note the volume of bismuth nitrate consumed (B).

Similarly pipette out 20.0 ml of standard EDTA (without adding sample aliquot) and titrate with standard bismuth nitrate (*see* 10.2.6) to the first formation of the yellow colour. Note the volume of bismuth nitrate consumed (A).

The difference in the volume of bismuth nitrate is corresponding to the amount of zirconia present in the sample. Calculate the percent by mass of  $ZrO_2$ . One ml of bismuth nitrate (1N) = 0.123 22 g of  $ZrO_2$ .

#### 10.5 Calculation

$$\text{Zirconia (ZrO}_2\text{) percent} = \frac{(A-B) C \times 0.123\ 22 \times 100}{M}$$

where

A = volume, in ml, of bismuth nitrate solution required to titrate the 20 ml of standard EDTA solution;

B = volume, in ml, of bismuth nitrate solution required to titrate the 20 ml of standard EDTA solution with 25 ml of sample aliquot;

C = strength of bismuth nitrate solution in molarity; and

M = mass, in g, of the sample in the aliquot.

NOTE—The mandelic acid procedure which is very specific for zirconium and hafnium precipitation is recommended instead of direct EDTA titration.

End point is not sharp as well as the zirconia values are low in direct EDTA titration. Hence back titration with bismuth nitrate using thiourea indicator is recommended as an alternate procedure.

#### 10.6 Reproducibility

Precision and accuracy of the method must be tested with International standard reference materials (SRM) following adopted method as stated above. Compare the values obtained with certified values of the elements. The two values should be as close as possible, for example certified values of British Chemical Standard 'No. 388 on Zircon' are as follows:

$ZrO_2$	– 66.2 percent,
$SiO_2$	– 32.7 percent,
$Al_2O_3$	– 0.3 percent,
$Fe_2O_3$	– 0.06 percent, and
$TiO_2$	– 0.25 percent.

### 11 DETERMINATION OF ALUMINA BY ATOMIC ABSORPTION SPECTROMETER

#### 11.1 Outline of the Method

The sample is fused with borax-sodium carbonate mixture and silica is determined by gravimetric method after dehydration of the decomposed sample in sulphuric acid. The clear filtrate after silica removal is analyzed for alumina by standard addition method using atomic absorption spectrometer.

#### 11.2 Reagents

**11.2.1 Standard Aluminium Solution** (1 ml = 1 mg Al) — Dissolve accurately weighed 0.250 g of aluminium metal powder purity 99.99 percent minimum in dilute nitric acid and make upto 250 ml in a standard volumetric flask maintaining nitric acid concentration to be 2 percent.

**11.2.2 Dilute Standard Aluminium Solution** (1 ml = 200 µg Al) — Dilute stock aluminium solution (*see* 11.2.1) five times to get aluminium (1 ml = 200 µg Al) maintaining nitric acid concentration to be 2 percent.

**11.2.3 Tartaric Acid**— 50 percent (m/v).

#### 11.3 Apparatus

**11.3.1 Atomic Absorption Spectrometer** — Equipped with a monochromatic radiation source such as aluminium hollow cathode lamp, a monochromator to isolate the 309.3 nm resonance line, an atomization

source such as a burner, nebulizer system and a read out device.

### 11.3.2 Operating Parameters

#### 11.3.2.1 Hollow cathode lamp, aluminium

#### 11.3.2.2 Wavelength, 309.3 nm

#### 11.3.2.3 Flame, N<sub>2</sub>O, acetylene.

NOTE— Other operating parameters to be followed according to manufacturer's instruction.

### 11.4 Procedure

**11.4.1** Find out approximate absorbance of the solution preserved (solution S) (see 6.3.5) at 309.3 nm line in N<sub>2</sub>O - C<sub>2</sub>H<sub>2</sub> flame. From this absorbance, the amount of sample aliquot to be taken for dilution and the range of aluminium standards to be added can be determined. The absorption of the sample aliquot and sample plus standards should lie in the linear portion of absorbance *versus* concentration graph. The samples, standards and blanks should be prepared to a fixed volume as per sequences of addition of various reagents shown in Table 1.

#### 11.4.2 Adjustment of the Atomic Absorption Spectrometer

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and the aluminium hollow cathode lamp. Fit the correct burner for nitrous oxide-acetylene flame and light the appropriate flame. Wait for about 20 min for stabilization. Set the wavelength at 309.3 nm, optimize instrument response by adjusting the wavelength, fuel, N<sub>2</sub>O burner head and nebulizer while aspirating the highest calibration solution.

As the sensitivity varies from instrument to instrument the concentration of the standard series and of the test solution should be adjusted accordingly. At the same time check the linearity of the calibration curve.

Aspirate water and one of the calibration solution repeatedly to ensure that there is no drift of absorbance. Finally aspirate water and set the absorbance to zero reading.

### 11.4.3 Atomic Absorption Measurement

**11.4.3.1** Aspirate first the diluted blank test solution, and then the calibration solution in increasing order, aspirating water between each aspiration of the solution and record the absorbance reading. Then aspirate like wise, the test sample and note the absorbance. Each aspiration should be made atleast three time, and take the average value. Solids which build up on the burner slit must be removed otherwise they will cause a decrease of sensitivity.

**11.4.3.2** Deduct the blank value from the absorbance of the standard as well as the test solution.

**11.4.3.3** Prepare a calibration curve by plotting the absorbance value of the standard solution against the concentration in µg/ml of the element or get the reading as computed from the computer.

**11.4.3.4** Compute the concentration of the test solution (corrected for the blank) to µg of the element per ml from the calibration curve.

### 11.5 Calculation

$$\text{Aluminium, percent by mass} = \frac{m \times 25 \times 250 \times 100}{M \times 20 \times 10^6}$$

where

$m$  = concentration, in g/ml, of the diluted sample solution after deducting the concentration of the diluted reagent blank solution; and

$M$  = mass, in g, of the sample taken.

**11.5.1** The percentage of Alumina shall be calculated as shown below:

$$\text{Alumina, percent} = \text{Aluminium percent} \times 1.889$$

**Table 1 Sequence of Addition of Various Reagents***(Clause 11.4.1)*

<b>Sample Solution</b>	<b>Reagent Blank Aliquot ml</b>	<b>Sample Aliquot ml</b>	<b>Aluminium Std 100 µg/ml</b>	<b>Volume of Concentrated <math>H_2SO_4</math> ml</b>	<b>Volume of Tartaric Acid 50 Percent (m/v) ml</b>	<b>Final to be Made ml</b>
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Sample blank	0.0	20.0	0.0	0.5	1	25.0
Std-1	0.0	20.0	0.5	0.5	1	25.0
Std-2	0.0	20.0	1.0	0.5	1	25.0
Std-3	0.0	20.0	1.5	0.5	1	25.0
Std-4	0.0	20.0	2.0	0.5	1	25.0
Reagent blank	20.0	0.0	0.0	0.5	1	25.0
Reagent blank + standard	20.0	0.0	1.0	0.5	1	25.0

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